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(54) **Method for spraying material particles in continous hydrothermal reaction and apparatus therefor**

Verfahren und Vorrichtung zur Sprühen von Feststoffteilchen in einer kontinuierlichen hydrothermalen Reaktion

Procédé et dispositif pour la pulvérisation de particules de substances solides dans une réaction hydrothermale en continu

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Description

BACKGROUND OF THE INVENTION

1. Field of the Invention

[0001] The present invention provides a spraying method and apparatus for spraying a starting material slurry through a nozzle in a continuous hydrothermal reaction, particularly under subcritical to supercritical conditions beyond the saturated vapor temperature to form fine particles without agglomeration thereof for efficiently effecting the hydrothermal reaction.

2. Description of the Prior Art

[0002] Since the rates of reactions of many oxides such as silica and alumina with high-pressure steam, particularly water having a temperature of at least the critical temperature, become fast in the presence of high-temperature water, particularly high-temperature and high-pressure water, hydrothermal synthesis reactions are widely carried out by making much of this phenomenon. A technique of hydrothermal synthesis of particles under pressurized conditions beyond the critical pressure (Tp = 218.3 atm) in a temperature range beyond the saturated vapor temperature, particularly beyond the critical temperature (e.g., Tc = 374°C for water), is carried out according to a batch method. In a large-scale batch method, however, a long heat-up time from the point of time when a starting material is set is taken together with a long temperature dropping time besides the period of time during which the desired temperature and pressure are maintained. Furthermore, there is a limit to efficient synthesis with one equipment because of batchwise production.

[0003] On the other hand, a method wherein a slurry is continuously fed through assembled piping for treatment therof is disclosed, for example, in Japanese Patent Laid-Open JP-A-5 305 7112 (1978), but is not aimed at a hydrothermal reaction of particles to be different in purpose from the present invention. Besides, Japanese Patent Publication JP-B-2 005 136 (1990) discloses a technology according to which a reaction is effected under the saturated vapor pressure. There are no continuous hydrothermal reaction technologies according to which a hydrothermal reaction of particles is effected with such assembled pipings as to maintain a pressurized state particularly at a temperature exceeding the critical temperature, followed by temperature drop and pressure reduction, and subsequent discharge of the reacted particles.

[0004] When a hydrothermal reaction is to be consistently effected in a continuous piping, there arise various difficulties such as a problem with feeding of a starting material slurry, a problem with retention of a piping in a state of being heated at a high temperature, and a problem with withdrawal of synthesized particles. The present invention is to solve a problem involved particularly in feeding a starting material slurry into a reaction chamber. More specifically, even if a starting material slurry is flowed in a piping in a temperature range beyond the saturated vapor temperature, particularly beyond the critical temperature, the solid in the slurry may possibly be separated to be stuffed in the piping because water is in the form of steam. In view of this, according to the present invention, the solid in the slurry is finely sprayed and then fed into a reaction chamber in order to efficiently effect a reaction thereof without the sliding being stuffed in a piping.

[0005] Further US 5 558,783 discloses an improved apparatus and method for initiating and sustaining an oxidation reaction wherein a fuel, such as natural gas or hazardous waste, is introduced into a reaction zone within a pressurized containment vessel. A permeable liner is placed within the containment vessel and around the reaction zone. An oxidizer, preferably oxygen, is mixed with a carrier fluid, such as water, and the mixture is heated and pressurized to supercritical conditions of temperature and pressure. This supercritical oxidizer carrier mix is introduced to the reaction zone gradually and uniformly, over a comparatively large area, by forcing it radially inward through the permeable liner and toward the reaction zone. The oxidizer carrier mixture is fed into the pressure vessel via an oxidizer and carrier fluid port, while a fuel/waste mixture is fed into the pressure vessel via a waste inlet port. Both of the carrier fluid/oxidizer mixture and the fuel/waste mixture are heated by respective heaters prior to being fed into the reactor vessel. Still further, Journal of the American Ceramic Society, Vol. 75, No. 4, pages 1019 to 1022 discloses hydrothermal synthesis of metal oxide fine particles by conveying an aqueous solution of a metal salt and distilled water through separate pipes and mixing these components with each other at an inlet of the reactor to cause a hydrothermal reaction.

SUMMARY OF THE INVENTION

[0006] The present invention provides a method for spraying starting material particles in a continuous hydrothermal reaction, as specified in appended claim 1.

[0007] The present invention also provides an apparatus for spraying starting material particles in a continuous hydrothermal reaction, as specified in appended claim 3.

BRIEF DESCRIPTION OF THE DRANING

[0008] The single figure is a cross-sectional view of an example of the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0009] The present invention is applicable to hydro-

thermal reactions of metal oxides such as alumina (Al_2O_3), zirconia (ZrO_2), choromia (Cr_2O_3), hafnia (HfO_2), and titania (TiO_2). The present invention will now be specifically illustrated based on the accompanying drawing.

Example

[0010] The drawing is an example of an apparatus used for carrying out the present invention. In the drawing, numeral 1 refers to an inlet for an aqueous fluid having a temperature exceeding the saturated vapor temperature, 2 to an inlet for a starting material slurry pressurized at ordinary temperature, and 3 to an ejection orifice for a mixture thereof. The ejection orifice 3 is constituted of a spray orifice 3-1 on the outer side thereof and a feed orifice 3-2 in a central portion thereof. Numeral 4 refers to a starting material feed pipe (feed piping), and 5 and 6 to double pipes surrounding the starting material slurry feed pipe 4, the inner pipe 5 of which is opened at the left end thereof to communicate with the outer pipe 6. Numeral 7 refers to a cooling medium inlet, and 8 to a cooling medium outlet. A cooling medium such as water or air is fed from the cooling medium inlet 7, passed along the outer side of the starting material slurry feed pipe 4 in the inner pipe 5 of the double pipes to reach the left end, turned back in the outer pipe 6, and discharged from the cooling medium outlet 8. During this course, the cooling medium inside the inner pipe 5 suppresses the heat-up of the starting material slurry feed pipe 4, and prevents the influence of heat of the aqueous fluid fed from the side of the outer pipe 6. The cooling medium passed through the outer pipe 6 absorbs heat of the aqueous fluid, and gives off the absorbed heat out of the system from the cooling medium outlet 8. The pressurized and heated aqueous fluid and the pressurized starting material slurry are effected from the mixture ejected orifice 3 and mixed with each other to reach the inside of a mixing chamber 9. During this course, particles in the starting material are finely pulverized simultaneously with separation of agglomerated particles.

[0011] Alternatively, a heat-insulating material may be packed instead of the double pipes, or a heat-insulating material itself may be fabricated into a hollow tube through which a cooling medium is passed.

[0012] According to the present invention, a starting material slurry is sprayed particularly under subcritical to supercritical conditions beyond the saturated vapor temperature to form fine particles without agglomeration thereof, whereby a hydrothermal reaction can be continuously and efficiently effected. Particularly, the starting material slurry, while separated from an aqueous fluid, is set at ordinary temperature just until the starting material slurry is mixed with the aqueous fluid, whereby troubles such as clogging of a piping are obviated to enable a continuous hydrothermal reaction to be efficiently effected.

Claims

1. A method for spraying starting material particles in a continuous hydrothermal reaction, comprising spraying an aqueous fluid pressurized and heated in excess of the saturated vapor temperature from an aqueous fluid spray orifice while spraying a starting material slurry comprising the starting material particles and pressurized at ordinary temperature from a a starting material slurry feed orifice which is provided in a central portion of the aqueous fluid spray orifice in which entry of heat into said starting material slurry from said aqueous fluid is avoided by a heat-insulating layer interposed between an aqueous fluid feed portion and a starting material slurry feed portion;

   and mixing with each other said starting material slurry and said aqueous fluid to reach the inside of a mixing chamber.

2. A method for spraying starting material particles in a continuous hydrothermal reaction as set forth in claim 1, wherein said heat-insulating layer is of a forced cooling system wherein a cooling medium is fed thereinto.

3. An apparatus for spraying starting material particles in a continuous hydrothermal reaction, wherein a feed orifice (3-2) for a starting material slurry comprising the starting material particles and pressurized at ordinary temperature is opened in a central portion of a spray orifice (3-1) for an aqueous fluid pressurized and heated in excess of the saturated vapor temperature, provided that a feed piping (4) for said starting material slurry is covered with a heat-insulating layer in order to avoid entry of heat into said slurry from the surrounding aqueous fluid heated in excess of the saturated vapor temperature, wherein said heat-insulating layer is constituted of double pipes (5, 6) surrounding said feed piping (4) for said starting material slurry, provided that a cooling medium is fed from a side of said double pipes on which side said starting material slurry is fed, and discharged via a side of said double pipes (5, 6) on which side said aqueous medium is fed.

Patentansprüche

1. Verfahren zum Sprühen von Ausgangsmaterialpartikeln bei einer kontinuierlichen hydrothermalen Reaktion, welches umfasst: Ein unter Druck stehendes wässriges Fluid, das auf eine Temperatur oberhalb der Sattdampftemperatur erhitzt wurde, wird aus einer Sprühöffnung für das wässrige Fluid herausgesprüht, während dabei ein Ausgangsmaterial-Schlicker, der die Ausgangsmaterialpartikel enthält, unter Druck steht und auf eine gewöhnliche

Temperatur erhitzt ist, aus einer Ausgangsmaterial-Schlicker-Zuführöffhung herausgesprüht wird, welche in einen mittigen Abschnitt der Sprühöffnung für das wässrige Fluid vorgesehen ist, wobei der Eintritt von Wärme aus dem wässrigen Fluid in den Ausgangsmaterial-Schlicker durch eine Wärmeisolationsschicht verhindert wird, die sich zwischen einem Zuführabschnitt für das wässrige Fluid und einem Ausgangsmaterial-Schlicker-Zuführabschnitt befindet;
und der Ausgangsmaterial-Schlicker und das wässrige Fluid bei Erreichen des Inneren der Mischkammer miteinander vermischt werden.

2. Verfahren zum Sprühen von Ausgangsmaterialpartikeln bei einer kontinuierlichen hydrothermalen Reaktion nach Anspruch 1, bei welchem es sich bei der Wärmeisolationsschicht um ein Zwangskühlungssystem handelt, dem ein Kühlmedium zugeführt wird.

3. Vorrichtung zum Sprühen von Ausgangsmaterialpartikeln in einer kontinuierlichen hydrothermalen Reaktion, bei welcher eine Zuführöffnung (3-2) für einen Ausgangsmaterial-Schlicker, welcher die Ausgangsmaterialpartikel enthält, unter Druck steht und auf eine gewöhnliche Temperatur erhitzt ist, sich in einem mittigen Abschnitt einer Sprühöffnung (3-1) für ein wässriges Fluid befindet, das unter Druck steht und auf eine Temperatur oberhalb der Sattdampftemperatur erhitzt ist, wobei das Zuführrohr (4) für den Ausgangsmaterial-Schlicker mit einer Wärmeisolationsschicht überzogen ist, um einen Eintritt von Wärme in den Schlicker vom umgebenden wässrigen Fluid her, das auf eine Temperatur oberhalb der Sattdampftemperatur erhitzt ist, zu verhindern, und die Wärmeisolationsschicht aus doppelten Rohren (5, 6) besteht, die das Zuführrohr (4) für den Ausgangsmaterial-Schlicker umgeben, und wobei ein Kühlmedium von derjenigen Seite der doppelten Rohre her zugeführt wird, von welcher der Ausgangsmaterial-Schlicker zugeführt wird, und über diejenige Seite der doppelten Rohre (5, 6) abgeführt wird, von welcher her das wässrige Medium zugeführt wird.

Revendications

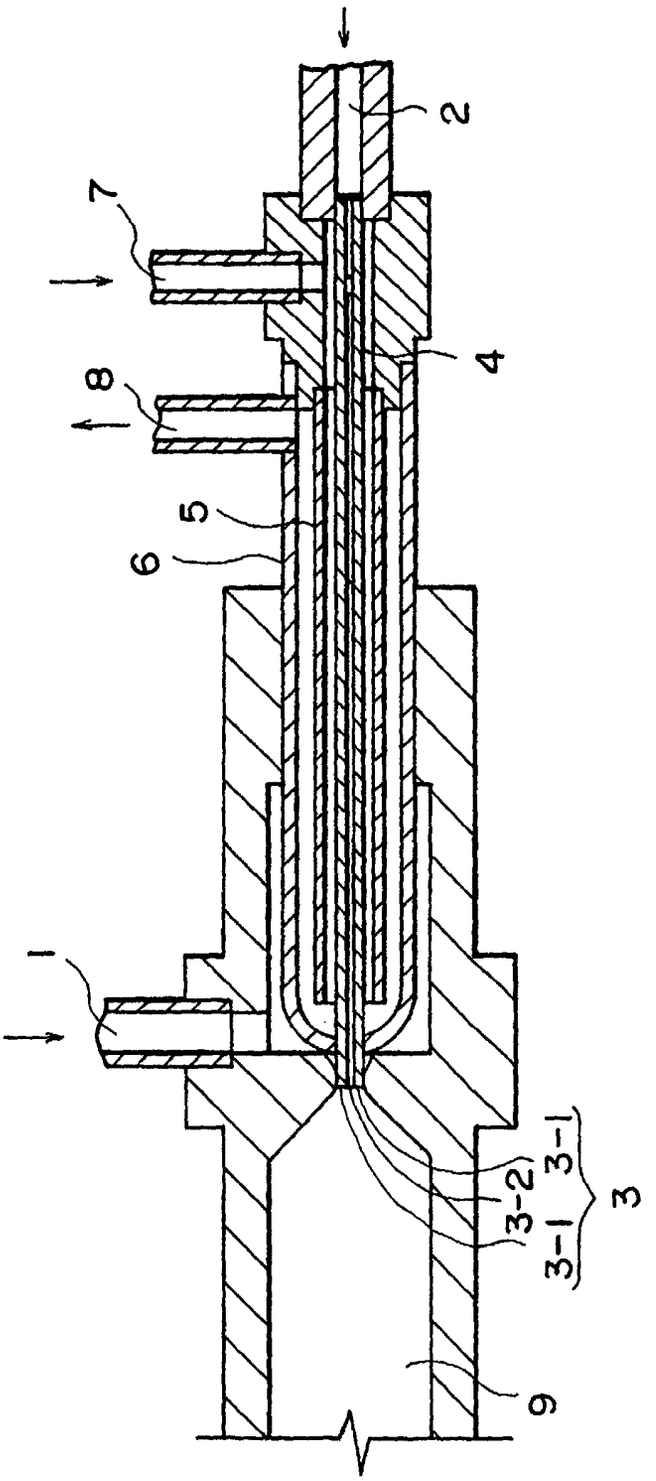
1. Procédé permettant d'introduire par pulvérisation une matière première à l'état de particules dans une réaction hydrothermique se déroulant en continu, lequel procédé comporte :

- le fait de pulvériser un fluide aqueux, pressurisé et chauffé à une température dépassant la température de vapeur saturante, en faisant passer ce fluide par un orifice de pulvérisation de fluide aqueux, tout en pulvérisant une suspension de matière première contenant la matière première à l'état de particules, pressurisée et à la température ordinaire, en faisant passer cette suspension par un orifice d'alimentation en suspension de matière première, lequel orifice est situé dans une partie centrale de l'orifice de pulvérisation de fluide aqueux, une couche thermo-isolante, interposée entre une zone pour alimentation en fluide aqueux et une zone pour alimentation en suspension de matière première, permettant d'éviter que de la chaleur soit transférée dudit fluide aqueux vers ladite suspension de matière première,
- et le fait de mélanger l'une avec l'autre ladite suspension de matière première et ledit fluide aqueux, à l'intérieur d'une chambre de mélange.

2. Procédé permettant d'introduire par pulvérisation une matière première à l'état de particules dans une réaction hydrothermique se déroulant en continu, conforme à la revendication 1, dans lequel ladite couche thermo-isolante fait partie d'un système de refroidissement à circulation entretenue dans lequel on fait passer un milieu réfrigérant.

3. Appareil servant à introduire par pulvérisation une matière première à l'état de particules dans une réaction hydrothermique se déroulant en continu, dans lequel :

- il y a un orifice d'alimentation (3-2), destiné à une suspension de matière première contenant la matière première à l'état de particules, pressurisée et à la température ordinaire, qui s'ouvre dans une partie centrale d'un orifice de pulvérisation (3-1), destiné à un fluide aqueux pressurisé et chauffé à une température dépassant la température de vapeur saturante,
- il y a une conduite d'alimentation (4), destinée à ladite suspension de matière première, qui est recouverte d'une couche thermo-isolante servant à éviter que de la chaleur soit transférée vers ladite suspension de matière première à partir dudit fluide aqueux qui entoure celle-ci et qui est chauffé à une température dépassant la température de vapeur saturante, ladite couche thermo-isolante étant constituée par une paire de tuyaux (5, 6) qui enveloppent ladite conduite d'alimentation (4) destinée à ladite suspension de matière première,
- et un milieu réfrigérant est introduit du côté desdits tuyaux où est introduite ladite suspension de matière première, puis évacué du côté desdits tuyaux (5, 6) où est introduit ledit fluide aqueux.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